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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Applicant:

GLOVER, JOHN N.

Filed: May 27, 1999

Art Unit: 1723

Application No.: 09/320,950

Examiner: David L. Sorkin

For: FILTERING MEDIUM AND
METHOD FOR CONTACTING SOLIDS
CONTAINING FEEDS FOR CHEMICAL
REACTORS

Docket No.: 20781.004

DECLARATION OF JOHN N. GLOVER

I, John N. Glover, declare that I am over the age of twenty-one (21) years of age and am fully competent to make this declaration. I have personal knowledge of the facts set forth in this declaration and they are true and correct. I declare:

1. I am the President of Crystaphase International, Inc. and its related corporate entities (hereinafter "Crystaphase"), and maintain an office at Crystaphase at 16825 Northchase Drive, Suite 660, Houston, TX. 77060-6029. I have been employed by Crystaphase since 1989 to the present as the President. I am the name inventor in the above-identified patent application and am familiar with the disclosure in the above-identified patent application.
2. I have worked in the petroleum refining and petrochemical industries for at least twenty-four years. I am familiar with ceramic filter units, catalysts, and recycling of these units.
3. I am a named inventor of the subject application and thus would be considered of above-ordinary skill in the art of ceramic filter units and associated methods. In my position of President, I have supervised numerous individuals and therefore am knowledgeable about the level of understanding of one with ordinary skill in the art in the field of ceramic filter units.
4. My educational experience includes undergraduate studies in Biology and Chemistry. I have performed numerous experiments on the subject matter of the above referenced patent application. I am extremely familiar with terms in the industry and the understanding associated with those terms throughout the industry.

5. I participated in an experiment in which comparative performance data was obtained for ceramic filter units comparing ceramic units in accordance with the present invention having combinations of elliptical and circular openings, along with flutes, to ceramic units in accordance with prior art units having combinations of circular openings and flutes. Five prior art ceramic units (Products A, B, C, D, and E) were compared to three ceramic units made in accordance with the present invention (Products F, G, and H, as shown in FIG. 4 of the present application).
6. Products A and B were spherical ceramic balls made in accordance with the ceramic units in U.S. Patent No. 4,615,796 issued to Kramer (hereinafter "Kramer"), with Product A having a 6" bed and Product B having a 12" bed.
7. Product C was a 5/8" disc with six circular openings and one central circular opening that is substantially similar to the closest prior art in "CE Refresher: Catalyst Engineering, Part 2" by John Fulton (hereinafter "Fulton") as shown at Fig. 1, third column, fifth row (hereinafter "Fulton Ceramic Unit"). A sample of Product C has been included and is labeled as C. Product C is manufactured by Haldor Topsoe A/S and is commercially available as TK-10. TK-10 has been on the market for approximately seventeen years and is the number one selling ceramic unit. Product C (i.e., TK-10) is the closest commercially available ceramic unit structurally to the Fulton Ceramic Unit. Product D is a 7/8" disc with six circular openings and one central circular opening. Product D is substantially similar to Product C, but with a larger diameter. To the best of my knowledge, the Fulton Ceramic Unit is not commercially available.
8. Product E is a 5/8" ceramic unit with one central circular opening and six flutes. Product E is commercially available as Dypor 607 and is manufactured by Dytech Corporation, Ltd. in Sheffield, England. A sample of Product E has been included and is labeled as E.
9. Product F is a 5/8" ceramic unit with one central circular opening and four surrounding elliptical openings made in accordance with the present invention. A sample of Product F has been included and is labeled as F. Product G is a 7/8" disc with one central circular opening and four surrounding elliptical openings, also made in accordance with the present

invention. Products F and G are commercially available as BG-1000 and are sold by the Assignee of the present invention.

10. Product H is a 7/8" elongated disc with one central circular opening and four surrounding elliptical openings made in accordance with the present invention. Product H is commercially available as BG-1002 and is sold by the Assignee of the present invention. A sample of Product H has been included and is labeled as H. Product H is twice as long as Product G.
11. A test apparatus was constructed using a 12" internal diameter by 18" tall 26 gauge steel cylinder with a collection grid inside the cylinder, as shown in FIG. 1 attached hereto. The collection grid was constructed of $\frac{1}{2}$ " thick grating on top of a solid plate, which was placed in the bottom of the cylinder as a collector floor, as shown in FIG. 3 attached hereto. The plate was drilled with 253 holes through the cells of the grating, each having a $\frac{1}{4}$ " diameter. Each one of the holes was centered in the collection grid with 0.65" centers, which created collection squares or cells, as shown in FIG. 3 attached hereto. The collection grid was secured to the floor using a silicon sealer.
12. Clear plastic tubes were pressed into each hole from below until the tubes extended approximately $\frac{1}{16}$ " above the top of the plate. A watertight seal was formed around each of the tubes. A clear plastic baffle was drilled to match the holes in the collector floor and installed $\frac{1}{2}$ " above the end of the 8" plastic tubes, as shown in FIG. 2 attached hereto. Both the collector and the lower portion of the plastic tubes were marked to accurately identify each individual tube during experimenting.
13. A single flow-regulated water inlet was installed so that the inlet could be accurately centered and placed six inches above the top of the bed to be tested. A six inch headspace is commonly used in trickle bed reactors into which the present invention is commonly installed. The water flow rate used in the experiments was one liter per minute.
14. The flow device and the steel cylinder/collector assembly were mounted on a seven foot tall stand. The fluid flow collection was at eye-level, where it could be easily observed.

15. A 1,000 mL graduated cylinder was used to collect and measure the flow through a single tube. A tight fitting funnel was placed over the cylinder to ensure that no water would enter other than through the single plastic tube. The funnel was slip-fitted over each collector tube one at a time. A digital timer was used for timing.
16. Several measurements were taken during the experiments to help determine the amount of lateral fluid distribution that was achieved using several different ceramic units. Table I summarizes the results of each experiment. The prior art ceramic unit results are shaded in gray in Table I and the results for the ceramic units made in accordance with the present invention are non-shaded and located on the right side of Table I.
17. The first measurement that was used to compare the lateral fluid distribution caused by the ceramic units was a determination of the number of cells that had liquid flow present within the collection grid. The larger the number of cells with flow, or active cells, indicates better lateral distribution because the feed stream is distributed across a larger area containing cells. The lower the flow rate within each cell also indicates better lateral distribution due to the dividing of the feed stream by the cells that distributes the feed stream better laterally. The results of this experiment are shown in Table I in the row labeled as "1. Total Number of Active Cells" and "2. % of Active Cells." The percentage of active cells is calculated by dividing the number of active cells by the total number of cells, 253. The best performing prior art ceramic unit was Product E. The best performing ceramic unit made in accordance with the present invention was Product F. Product F had 11% more active cells than the best performing prior art ceramic unit in this experiment, which represents a 46% improvement over the prior art.
18. The next experiment that was conducted determined an active area of the grid in which flow was determined and is labeled as the row "3. Area of Active Cells". The larger the Area of Active Cells, the better. The larger Area of Active Cells indicates better lateral distribution than a smaller Area of Active Cells. The Area of Active Cells was calculated by multiplying the horizontal distance of the active cells by the vertical distance of the active cells. Not every cell within the Area of Active Cells has flow. The ceramic unit made in accordance with the present invention labeled in Table I as Product F performed the best with the greatest Area of Active Cells being 180. The prior art ceramic unit labeled as Product C in

Table I performed the best with 143 active cells. It is believed that Product C would perform better than the Fulton Ceramic Unit because Product C has more openings than the Fulton Ceramic Unit. Product F made in accordance with the present invention performed approximately 26% better than the prior art Product C in this experiment.

19. Measurements were taken to determine the distance the flow was laterally distributed based upon the feed location. Product H, which is made in accordance with the present invention, performed the best compared to any of the tested ceramic units, with a total of ten cells with flow located greater than five cells away from the central feed location and three cells with flow located greater than six cells away from the central feed location. Out of the prior art ceramic units that were tested, the best performance was obtained by using Product C. Product C only had two cells with flow located greater than five cells away from the central feed location. No cells greater than six cells away from the central feed location had any flow in them in the prior art ceramic units. Product H performed at least five times better than Product C when determining the number of active cells greater than five cells away from the feed stream location. Product H performed at least three times better than Product C when determining the number of active cells greater than six cells away from the feed stream location.
20. Measurements were also taken of the flow rates within each cell. A lower flow rate is indicative of better lateral distribution, since the flow is distributed across a larger number of cells. The present invention embodiments with one central opening and surrounding elliptical openings consistently outperformed the prior art units tested.
21. The average flow rate per active cell was determined for each active cell. To determine this average flow rate, the total inlet feed flow rate was divided by the number of active cells. The lower the average flow rate, the better. A lower average flow rate per active cell indicates that the feed stream was distributed among a greater number of active cells. Product F performed the best with only 1.16% average of the flow rate. With respect to the prior art ceramic units, Product E performed the best with 1.69% average of the flow rate. The prior art with the closest structural similarity to the Fulton Ceramic Unit, Product C, had a 1.72% average of the flow rate. The present invention performed approximately 30% better than the best performing prior art ceramic units tested.

22. The maximum flow rate in a cell was also measured for all of the tested ceramic units. The maximum flow in a cell was determined by measuring the flow rates of each active cell and determining the highest flow rate of those cells. In this experiment, the lower the maximum flow rate, the better. The best performing ceramic unit tested was Product F with only a 4.46% maximum flow rate in any one cell. The best performing prior art ceramic unit was Product C with an 8.45% maximum flow rate in any one cell. The best embodiment of the present invention, Product F, performed approximately 47% better than the best performing prior art ceramic unit tested, Product C.
23. Measurements for the percentage of active cells with greater than 3% of total flow and greater than 5% of total flow were also taken. The percentage of active cells with greater than three and five percent of the total flow was determined by comparing the flow rates of the active cells with three and five percent of the total flow rate of the inlet feed stream respectively. With respect to the experiment measuring greater than 3% of total flow, the best performer in accordance with the present invention was Product H with only 8.33% of the cells having a flow rate greater than 3% of the total flow rate. The best performing prior art was Product C with 17.24% of the cells having a flow rate greater than 3% of the total flow rate. In this experiment, the lower the percentage of active cells with greater than 3% of total flow, the better. The present invention, Product H, performed approximately 52% better than the prior art ceramic units, Product C, in this experiment. With respect to the experiment measuring greater than 5% of total flow, the best performer in accordance with the present invention was Product H with 0% of the cells having a flow rate greater than 5% of the total flow rate. The best performing prior art was the Product E with 5.08% of the cells having a flow rate greater than 5% of the total flow rate. In this experiment, the lower the percentage of active cells with greater than 5% of total flow, the better. The present invention, Product C, performed significantly better than the prior art ceramic units, Product E, in this experiment also.
24. To the best of my knowledge and understanding, based upon experiments that I performed, lateral fluid distribution was improved in all performance indicators measured when using the ceramic units of the present invention compared with use of prior art ceramic units.

Product F performed the best consistently when compared with the consistently best performing prior art ceramic filter unit, Product C.

25. The attached Table I demonstrates the amount of lateral fluid distribution that was obtained by using the ceramics of the present invention and prior art ceramic units. As can be seen from the Table I, advantageous properties are associated with the use of the central opening with elliptical openings. The advantageous properties resulting from the use of elliptical openings are unexpected.
26. Crystaphase has enjoyed much commercial success from the sale of these ceramic units. Crystaphase began selling the ceramic units made in accordance with the present invention in 1998. Since then, Crystaphase has sold more than eight million dollars worth of units made in accordance with the present invention, which approximates 40,000 cubic feet of product being sold, which correlates to about 30% – 35% of the total market over the past six years. With so many units sold, the ceramic units should be deemed to have met an unfilled need in the industries in which these ceramic units have been sold.
27. I believe there is no motivation for one of ordinary skill in the field of ceramic filter units to utilize ceramic disc units containing a central circular opening and at least three elliptical openings in accordance with the present invention, at least without resorting to hindsight after viewing the present invention.
28. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Sec. 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the publication or any patent issued thereon.

Date

11/5/03

John N. Glover

TABLE I - SUMMARY OF COLD FLOW EXPERIMENT RESULTS

Shape	PRIOR ART				PRESENT INVENTION			
	Spheres		Cylindrical Openings		Elliptical Openings			
Product	A (3/4" Ceramic balls)	B (3/4" Ceramic balls)	C (5/8" TK-10)	D (7/8" TK-10)	E (5/8" Dypor 607)	F (5/8" BG-1000)	G (7/8" BG-1000)	H (7/8" BG-1002)
Top layer – Depth	6"	12"	6"	6"	6"	6"	6"	6"
Shape	Sphere	Sphere	Disc with 7 cylindrical openings	Disc with 7 cylindrical openings	Disc with one cylindrical opening and six flutes	Disc with four elliptical and one central circular openings	Disc with four elliptical and one central cylindrical openings	Elongated Disc with four elliptical and one central cylindrical openings
Void space	n/a	n/a	55%	55%	60%	60%	60%	63%
Bottom layer – Depth	6"	-	6"	6"	6"	6"	6"	6"
Size and Shape	3/4" Sphere	-	3/4" Sphere	3/4" Sphere	3/4" Sphere	3/4" Sphere	3/4" Sphere	3/4" Sphere
Void space	~39 %	-	~39 %	~39 %	~39 %	~39 %	~39 %	~39 %
1. Total number of active cells	36	46	58	46	59	86	69	84
2. % of active cells	14.23%	18.18%	22.92%	18.18%	23.32%	33.99%	27.27%	33.20%
3. Area of Active Cells	49	100	143	72	120	180	121	153
4. Number of active cells greater than 5 cells distance from center	0	0	2	0	1	4	2	10
5. Number of active cells greater than 6 cells distance from center	0	0	0	0	0	0	0	3
6. Average Flow Rate per Active Cell	2.78%	2.17%	1.72%	2.17%	1.69%	1.16%	1.45%	1.19%
7. Maximum Flow Rate in a Cell	10.42%	7.03%	8.45%	10.39%	9.07%	4.46%	7.17%	9.74%
8. Percentage of active cells greater than 3% of total flow	27.78%	23.91%	17.24%	26.09%	23.73%	10.47%	8.70%	8.33%
9. Percentage of active cells greater than 5% of total flow	25.00%	8.70%	5.17%	6.52%	5.08%	0.00%	7.25%	3.57%



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Steel Cylinder

Collection tubes beneath collection grid (not shown)

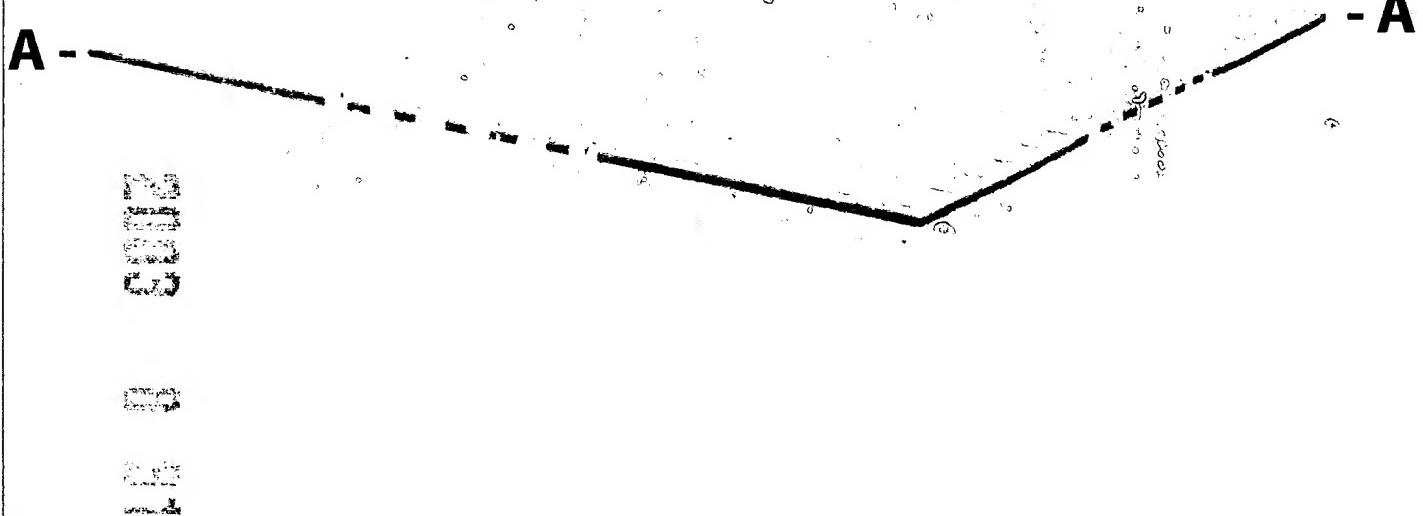
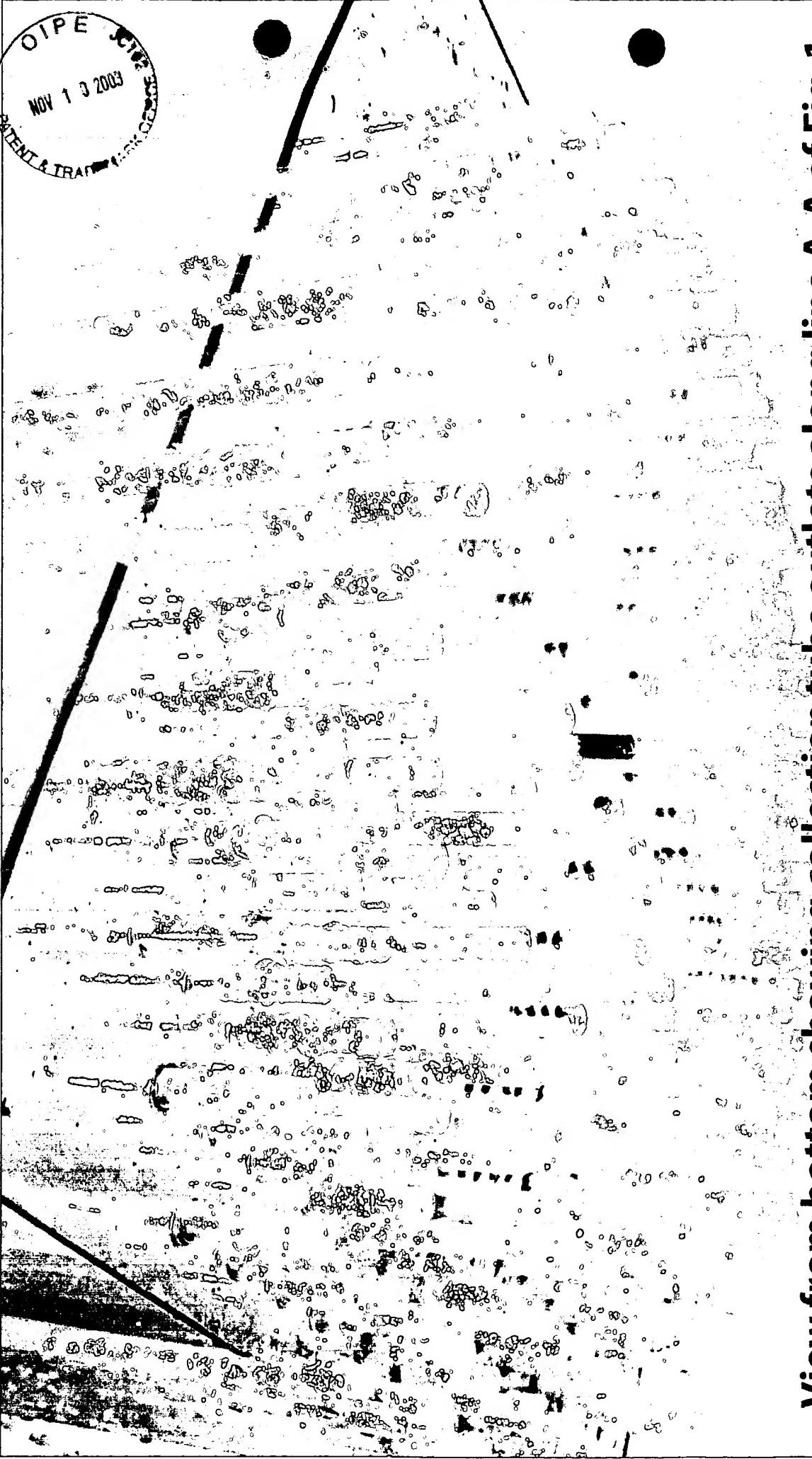
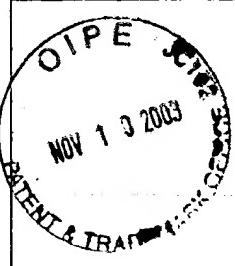


Fig. 1

S.N.09/320.950



View from bottom showing collection tube outlets along line A-A of Fig. 1

Fig. 2

S.N. 09/320,950

S.N. 09/320,950

Fig. 3

Closeup showing collector grid at bottom of steel cylinder

Individual collection cell -

